

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Stoyanov et. al.

Attorney Docket No. 25277

Application No. 10/748,930

Group Art Unit: 1731

Filed: 12/30/03

Examiner: Cordray, Dennis R.

Title: Individualized Intrafiber Crosslinked Cellulosic Fiber With Improved Brightness and Color

DECLARATION OF ANGEL STOYANOV PURSUANT TO § 37 C.F.R. § 1.132

Federal Way, WA,  
September 29, 2006

TO THE COMMISSIONER OF PATENTS:

I, Angel Stoyanov, declare and state as follows:

1. I am currently employed by the Weyerhaeuser Company as a Scientist and since 1998 have worked exclusively on crosslinking of cellulosic fibers.

2. I received my Bachelor of Science and my Master Of Science from the University of Chemical Technology and Metallurgy at Sofia, Bulgaria, in 1980 and 1981, respectively. After graduation my work history is as follows:

I was a Research Assistant from 1982 to 1986 and an Assistant Professor from 1986 to 1994 at the University of Chemical Technology and Metallurgy at Sofia, Bulgaria. From 1990 to 1991 I worked under a Fulbright scholarship at the University of Washington, Seattle, WA, and completed all graduate courses for a Ph. D. in 1996. From

1996 to 1998 I conducted research for my Ph. D. and held various teaching positions in the Department of Engineering at the University of Washington.

3. I have read and am familiar with the Hansen et al patent US Patent No. 6,340,411

4. Hansen et al state in the '411 patent that initial application of the binder on high bulk fibers preferably occurs after the curing step, particularly if the binder is capable of functioning as a crosslinking material. Hansen then states that specific binders that can also crosslink are polyols, polyaldehydes, polycarboxylic acids and polyamines. If such binders are present during curing, the binder will be consumed during the curing step to form covalently crosslinked bonds. When this occurs, the binder is no longer available for hydrogen bonding or coordinate covalent bonding, and particle binding to fibers is ineffective, column 34, line 1-13.

5. Tests were undertaken to determine if polyols indeed act as crosslinking agents with cellulose. Accordingly, I planned and supervised experiments which were carried out by my technician Derik Rieger.

6. Exhibit A shows the experimental design for the tests. All samples were cured at 171°C for 7 minutes. The acronyms are as follows: COP, chemical on pulp (CF416 pulp from Weyerhaeuser Co.); SHP, sodium hypophosphite; CA, citric acid; SOR, sorbitol; and XYL, xylitol. Exhibit B shows the addition levels for the various reagents; Exhibit C gives the procedure, Exhibit D shows the results of brightness testing by TAPPI T 525 om-02 and Exhibit E, the FAQ wet bulk results determined by the procedure in the application. The Hunter color values were determined by TAPPI T 1231 sp 98. Whiteness Index,  $WI_{(CDM-L)}$ , was calculated from the formula,  $WI_{(CDM-L)} = (L - 3b)$ .

7. The results are summarized in Table 1.

Table 1

## Fiber Properties

Sample	Wt. % on Dry Fiber				FAQ Wet Bulk, cc/g	ISO Brightness %	Hunter Color			WI <sub>(CDM-L)</sub>
	CA	SHP	Sorbitol	Xylitol			L	a	b	
A	0	0	0	0	11.59	82.7	94.9	-0.83	5.58	78.16
B	0	2	0	0	12.26	82.8	95.0	-0.83	5.58	77.87
C	8	2	0	0	18.48	78.5	94.7	-2.02	8.67	68.69
D	8	2	2	0	18.29	83.7	95.3	-1.41	5.53	78.71
E	8	2	6	0	17.05	85.4	95.7	-1.23	4.80	81.3
F	8	2	0	2	18.18	84	95.6	-1.45	5.7	78.50
G	8	2	0	6	16.83	85.8	95.7	-1.21	4.53	82.10
H	0	2	2	0	11.43	82.3	94.8	-0.88	5.81	77.37
I	0	2	6	0	11.10	81.4	94.4	-0.81	5.96	76.52
J	0	2	0	2	11.27	80.5	94.1	-0.78	6.20	75.50
K	0	2	0	6	10.76	79.8	93.3	-0.76	5.60	76.50

8. It is well recognized by those skilled in the art of crosslinked fibers that an increase in FAQ wet bulk, relative to an untreated control, reflects that fibers have been crosslinked.

9. Sample A is a control and Sample B is the pulp with 2 percent by dry weight sodium hypophosphite; FAQ wet bulk values are 11.59 and 12.26 cc/g, respectively, and  $WI_{(CDM-L)}$  values are 78.16 and 77.87, respectively. When pulp is treated with citric acid and sodium hypophosphite, Sample C, FAQ wet bulk is 18.48 cc/g and the Whiteness Index is 68.69. When pulp is treated with citric acid, sodium hypophosphite and sorbitol, a polyol, at the 2 and 6 percent by weight level of sorbitol on pulp, Samples D and E, respectively, FAQ wet bulk is significantly increased to 18.29 and 17.05 cc/g, respectively. The Whiteness Index of Samples D and E, also increased to 78.71 and 81.30, respectively. However, when pulp is treated only with sodium hypophosphite and two different levels of sorbitol, 2 and 6 percent by weight, Samples H and I, there is no increase in FAQ wet bulk; Whiteness Index, decreased relative to the control pulp and the pulp sample with only sodium hypophosphite, Samples A and B, respectively.

When pulp is treated with citric acid, sodium hypophosphite and xylitol, a polyol, at the 2 and 6 percent by weight level of xylitol on pulp, Samples F and G, respectively, FAQ wet bulk is significantly increased to 18.18 and 16.83 cc/g, respectively. The Whiteness Index of Samples F and G, also increased to 78.50 and 82.10, respectively. However, when pulp is treated only with sodium hypophosphite and two different levels of xylitol, 2 and 6 percent by weight, Samples J and K, there is no increase in FAQ wet bulk; Whiteness Index  $WI_{(CDM-L)}$ , decreased relative to the control pulp and the pulp with only sodium hypophosphite, Samples A and B, respectively.

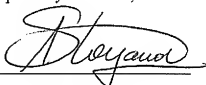
10. Based on the fact that there is no increase in FAQ wet bulk when pulp is treated only with sodium hypophosphite and sorbitol, or only with sodium hypophosphite and xylitol, it is my opinion that the polyol, sorbitol, and the polyol, xylitol, do not crosslink with cellulose.

11. In accordance with accepted Patent Office Practice, the dates in the laboratory notebook pages presented in Exhibits A- E have been redacted.

12. I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application of any patent issued therefrom.

Date 9/29/06

Respectfully submitted,

A handwritten signature in cursive script, appearing to read "Stoyanov", is written over a horizontal line.

Angel Stoyanov

## EXHIBIT A

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Project No. \_\_\_\_\_  
Book No. 14680TITLE Expt # 145 Solutions

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## Weyerhaeuser Confidential

## Patent Action

**Title:** Experiment # 145: CA + Polyols for Patent action**Objective(s):** Investigate whether polyols will be involved in crosslinking of cellulose fibers under the conditions used for esterification of cellulose with CA**Materials:**

- Pulp: CF416 - 94%
- Sample size: 20 g
- XLinker: CA
- Catalyst: SHP 9%
- Polyols: Sorbitol (Sorbidex) and Xylitol (Xylidex) 9%
- Fiberizer: 6" pad former
- Dispatch oven
- Metal baskets for curing

**Experimental Design:**

Sample ID	Chemistry	XLinker (% COP)	SHP (% COP)	Polyol		Cure Temp. (°F)	Cure time (min.)
				Sorbitol	Xylitol		
	-			(% COP)			
A	Blank	0	0	0	0	340	7
B	Pulp+SHP	0	2	0	0	340	7
C	CA+SHP	8	2	0	0	340	7
D	CA+SHP+SOR	8	2	2	0	340	7
E	CA+SHP+SOR	8	2	6	0	340	7
F	CA+SHP+XYL	8	2	0	2	340	7
G	CA+SHP+XYL	8	2	0	6	340	7
H	SHP+SOR	0	2	2	0	340	7
I	SHP+SOR	0	2	6	0	340	7
J	SHP+XYL	0	2	0	2	340	7
K	SHP+XYL	0	2	0	6	340	7

**Procedure:**

1. Weigh the sample 20 g (odb);
2. Apply the crosslinking solution using the usual syringe method;
3. Leave the samples overnight in a sealed plastic bags;
4. Use the 6" pad former for fluffing (50% consistency);
5. Cure the samples in the Dispatch V Series oven;
6. Store the cured fibers in a plastic bag.

**Testing:**

1. AFAQ Wet Bulk at 0.6 kPa
2. Brightness/Color

Witnessed &amp; Understood by me, \_\_\_\_\_ Date \_\_\_\_\_

Invented by \_\_\_\_\_

Recorded by \_\_\_\_\_

Date \_\_\_\_\_

## EXHIBIT B

TITLE Expt 145 SolutionsProject No. \_\_\_\_\_  
Book No. 14680

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From Page No. \_\_\_\_\_

**Exp# 145:CA+ Polyols for patent action**Date: \_\_\_\_\_  
Pulp CF418

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
<b>A</b>	CA	0	20	100	0.000	
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	0	20	1.20	0.000	

pH 7.15

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
<b>B</b>	CA	0	20	100	0.000	
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.483

pH 7.06

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
<b>C</b>	CA	8	20	100	1.800	1.597
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.482

pH 1.96

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
<b>D</b>	CA	8	20	100	1.800	1.603
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.479
	Sorbitol	2	20	100	0.400	0.401

pH 1.91

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
<b>E</b>	CA	8	20	100	1.800	1.603
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.480
	Sorbitol	2	20	100	1.200	1.202

pH 1.93

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
<b>F</b>	CA	8	20	100	1.800	1.605
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.480
	Xylitol	2	20	100	0.400	0.400

pH 1.92

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
<b>G</b>	CA	8	20	100	1.800	1.601
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.481
	Xylitol	6	20	100	1.200	1.191

pH 1.94

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
<b>H</b>	Sorbitol	2	20	100	0.400	0.399
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.485

pH 4.73

Witnessed &amp; Understood by me,

Date

Invented by

D. J. D. J. D. J.

Respected by

Date

To Page No. \_\_\_\_\_

## EXHIBIT B

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Project No.  
Book No. 19680

TITLE 8p4 14S Solution NATA

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Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
I	Sorbitol	6	20	100	1.200	1.202
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.482

pH 4.72

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
J	Xylitol	2	20	100	0.400	0.401
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.489

pH 4.75

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
K	Xylitol	6	20	100	1.200	1.199
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.484

pH 4.75

To Page No.

Witnessed &amp; Understood by me,

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Recorded by

Date



## EXHIBIT C

TITLE Exp #145: CA + Polyols for Patent action Project No. 14650  
Book No. 14650

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Substance

CF410 Pulp used - 20g @ 94% consistency = 21.28g pulp

(TARGET weight(g)) = Actual weight(g) Pulp + Solution (g)

21.20	21.24	40.88
21.28g	21.15	41.06
	21.21	41.15
	21.30	41.26
	21.34	41.31
	21.23	41.33
	21.37	41.17
	21.15	41.03
	21.34	41.13
	21.25	40.98
	21.22	41.13

- Prepared solutions on
- Applied to sheets
- Fiberized on            - Visual on fibres appears to be no different between samples, pre-curing.
- Samples air dry on table top for 4 hours before curing.
- Samples cured @ 340° for 7 min each on
- Samples placed in 50X humidity room before FAQ testing,
- TESTED: Brightness + color on
- FAQ TESTER in METZGER NOT used after many controls would NOT come into specs.
- FAQ TESTER in 116 was used

Witnessed & Understood by me,           Date           Invented by           Recorded by           Date           To Page No.

## EXHIBIT D

Project No.

Book No. 14660

TITLE Exp# 145

Brightness Results

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Exp#	Sample#	side	position	Operator	TEST DATE	BRIGHTNESS	R(X)	R(Y)	R(Z)	X	Y	Z	L	a
145	A	a	1	D	08/03/06	82.44	91.01	88.69	82.21	87.52	88.09	97.2	94.7	-0.84
145	A	a	2		08/03/06	82.42	91	89.85	82.18	87.5	88.05	97.16	94.69	-0.8
145	A	a	3		08/03/06	82.39	90.98	89.64	82.14	87.48	88.04	97.12	94.68	-0.82
145	A	b	1		08/03/06	83.03	91.79	90.41	82.76	88.24	90.41	97.85	95.08	-0.81
145	A	b	2		08/03/06	83.05	91.8	90.44	82.79	88.25	90.44	97.88	95.1	-0.85
145	A	b	3		08/03/06	83.04	91.77	90.42	82.78	88.23	90.42	97.87	95.09	-0.85
					Average	82.7	91.4	90.0	82.5	87.9	88.0	97.5	94.9	-0.8
					StDev	0.3	0.4	0.4	0.3	0.4	0.4	0.4	0.2	0.0
145	B	a	1	D	08/03/06	81.85	91.13	89.68	81.58	87.49	88.88	96.45	94.7	-0.88
145	B	a	2		08/03/06	81.87	91.05	89.58	81.42	87.39	88.58	96.27	94.85	-0.87
145	B	a	3		08/03/06	81.87	91.07	89.59	81.4	87.41	88.59	96.24	94.85	-0.87
145	B	b	1		08/03/06	83.8	92.16	90.92	83.57	88.09	90.02	98.81	95.35	-0.91
145	B	b	2		08/03/06	83.82	92.2	90.94	83.57	88.72	90.94	98.81	95.36	-0.88
145	B	b	3		08/03/06	83.79	92.15	90.89	83.55	88.68	90.89	98.79	95.34	-0.87
					Average	82.8	91.8	90.3	82.5	88.1	90.3	97.6	95.0	-0.9
					StDev	1.1	0.6	0.7	1.2	0.7	0.7	1.4	0.4	0.0
145	C	a	1	D	08/03/06	78.52	91.12	89.54	77.97	88.77	88.54	92.19	94.3	-1.08
145	C	a	2		08/03/06	78.54	91.12	89.58	77.98	88.77	88.56	92.2	94.3	-2
145	C	a	3		08/03/06	78.56	91.19	89.63	78.02	88.83	89.83	92.25	94.87	-2.02
145	C	b	1		08/03/06	78.29	91.2	89.59	77.72	88.79	89.59	91.89	94.05	-2.03
145	C	b	2		08/03/06	78.61	91.57	89.93	78.02	87.13	88.93	92.24	94.83	-2.02
145	C	b	3		08/03/06	78.87	91.53	89.87	78.07	87.11	88.92	92.31	94.83	-2.04
					Average	78.5	91.3	89.7	78.0	86.9	88.7	92.2	94.7	-2.0
					StDev	0.1	0.2	0.2	0.1	0.2	0.2	0.1	0.1	0.0
145	D	a	1	D	08/03/06	83.04	91.97	91.05	83.47	88.52	91.05	98.06	95.42	-1.48
145	D	a	2		08/03/06	84.11	92.19	91.28	83.7	88.74	91.28	98.06	95.54	-1.48
145	D	a	3		08/03/06	84.26	92.33	91.37	83.86	88.86	91.37	98.15	95.59	-1.38
145	D	b	1		08/03/06	83.29	91.33	90.38	82.88	87.9	90.38	98	95.07	-1.38
145	D	b	2		08/03/06	83.35	91.41	90.45	82.94	87.98	90.45	98.06	95.1	-1.37
145	D	b	3		08/03/06	83.5	91.52	90.59	83.09	88.09	90.59	98.24	95.18	-1.42
					Average	83.7	91.8	90.9	83.3	88.4	90.9	98.5	95.3	-1.4
					StDev	0.4	0.4	0.4	0.4	0.4	0.4	0.5	0.2	0.0
145	E	a	1	D	08/03/06	85.07	92.18	91.38	84.78	88.94	91.39	100.23	95.8	-1.28
145	E	a	2		08/03/06	85.52	92.57	91.75	85.19	89.33	91.75	100.72	95.78	-1.22
145	E	a	3		08/03/06	85.63	92.63	91.8	85.28	89.39	91.8	100.81	95.81	-1.19
145	E	b	1		08/03/06	85.11	92.18	91.37	84.81	88.93	91.37	100.27	95.59	-1.28
145	E	b	2		08/03/06	85.34	92.42	91.6	85	89.17	91.6	100.5	95.71	-1.23
145	E	b	3		08/03/06	85.47	92.69	91.88	85.36	89.46	91.88	100.92	95.86	-1.22
					Average	85.4	92.4	91.6	85.1	89.2	91.6	100.8	95.7	-1.2
					StDev	0.3	0.2	0.2	0.2	0.2	0.2	0.3	0.1	0.0
145	F	a	1	D	08/03/06	83.6	92.07	91.08	83.22	88.55	91.08	98.39	95.44	-1.45
145	F	a	2		08/03/06	83.91	92.35	91.34	83.48	88.82	91.34	98.71	95.57	-1.43
145	F	a	3		08/03/06	83.04	92.38	91.39	83.49	88.85	91.39	98.71	95.8	-1.48
145	F	b	1		08/03/06	83.99	92.24	91.3	83.6	88.78	91.3	98.85	95.55	-1.47
145	F	b	2		08/03/06	84.17	92.4	91.43	83.73	88.91	91.43	98	95.62	-1.43
145	F	b	3		08/03/06	84.09	92.31	91.38	83.69	88.83	91.38	98.95	95.59	-1.48
					Average	84.0	92.3	91.3	83.5	88.8	91.3	98.8	95.6	-1.5
					StDev	0.2	0.1	0.1	0.2	0.1	0.1	0.2	0.1	0.0
145	G	a	1	D	08/03/06	85.84	92.28	91.54	85.38	89.12	91.54	100.92	95.88	-1.23
145	G	a	2		08/03/06	86.08	92.65	91.9	85.74	89.5	91.9	101.38	95.88	-1.17
145	G	a	3		08/03/06	86.04	92.67	91.89	85.74	89.51	91.89	101.37	95.88	-1.14
145	G	b	1		08/03/06	85.86	92.48	91.71	85.53	89.31	91.71	101.13	95.77	-1.19
145	G	b	2		08/03/06	85.86	92.3	91.55	85.29	89.14	91.55	100.85	95.68	-1.21
145	G	b	3		08/03/06	85.47	92.13	91.43	85.18	88.98	91.43	100.69	95.82	-1.3
					Average	85.8	92.4	91.7	85.5	89.3	91.7	101.1	95.7	-1.2
					StDev	0.2	0.2	0.2	0.2	0.2	0.2	0.3	0.1	0.1
145	H	a	1	D	08/03/06	82.22	91.17	89.78	81.98	87.59	88.78	96.87	94.75	-0.88
145	H	a	2		08/03/06	82.22	91.15	89.75	81.95	87.57	88.75	96.87	94.74	-0.85
145	H	a	3		08/03/06	82.17	91.09	89.72	81.87	87.51	88.72	96.79	94.72	-0.9
145	H	b	1		08/03/06	82.43	91.38	89.97	82.12	87.78	89.97	97.09	94.85	-0.88
145	H	b	2		08/03/06	82.35	91.31	89.93	82.05	87.72	89.93	97.01	94.83	-0.89
145	H	b	3		08/03/06	82.28	91.29	89.9	81.97	87.68	89.9	96.91	94.81	-0.89
					Average	82.3	91.2	89.8	82.0	87.6	89.8	96.9	94.8	-0.9
					StDev	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.0

To Page No.

Witnessed &amp; Understood by me, ...

Date

Invented by

Recorded by

Date

TITLE Expt# 145 Brightness Results

Book No. 14660

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From Page No.

HUNTER WISNIEWICZ WIECIST												
5.32	95.67	-0.81	5.51	0	0	575.06	5.25	89.89	50.6	64.42	-1.83	
5.52	95.85	-0.77	5.51	0	0	575.15	5.28	89.85	50.72	64.38	-1.90	
5.54	95.85	-0.79	5.54	0	0	575.11	5.28	89.84	50.64	64.27	-1.97	
5.63	96.17	-0.78	5.62	0	0	575.10	5.34	90.41	50.81	64.74	-2.03	
5.83	96.18	-0.82	5.82	0	0	575.07	5.34	90.44	50.83	64.77	-1.97	
5.62	96.17	-0.82	5.81	0	0	575.05	5.33	90.42	50.86	64.70	-1.96	
5.0	96.0	-0.8	5.6	0.0	0.0	575.1	5.3	90.0	50.8	64.6	-1.9	
0.1	0.2	0.0	0.1	0.0	0.0	0.0	0.0	0.4	0.1	0.2	0.0	
5.93	95.81	-0.85	5.99	0	0	575.12	5.71	89.68	57.27	62.21	-2.14	
5.83	95.82	-0.84	6.04	0	0	575.17	5.76	89.58	56.96	61.89	-2.19	
6.08	95.83	-0.84	6.06	0	0	575.18	5.79	89.59	56.83	61.77	-2.22	
5.4	96.38	-0.87	5.37	0	0	574.8	5.08	90.02	61.51	68.42	-1.72	
5.41	96.39	-0.85	5.38	0	0	574.87	5.09	90.04	61.48	68.39	-1.77	
5.39	96.37	-0.84	5.38	0	0	574.89	5.07	90.06	61.58	68.43	-1.70	
5.7	96.1	-0.8	5.7	0.0	0.0	575.0	5.4	90.3	59.3	64.2	-2.0	
0.3	0.3	0.0	0.4	0.0	0.0	0.0	0.2	0.4	0.7	2.5	2.4	
8.50	95.81	-1.92	8.00	0	0	573.8	8.17	89.54	43.27	49.58	-1.85	
8.58	95.81	-1.94	8.00	0	0	573.75	8.17	89.56	43.27	49.59	-1.8	
8.58	95.84	-1.96	8.71	0	0	573.72	8.19	89.03	43.2	49.57	-1.78	
8.78	95.82	-1.97	8.92	0	0	573.79	8.39	89.50	42.12	48.57	-1.86	
8.79	95.87	-1.96	8.93	0	0	573.82	8.39	89.51	42.28	48.91	-1.91	
8.75	95.98	-1.97	8.88	0	0	573.77	8.34	89.57	42.53	49.12	-1.85	
5.7	95.9	-2.0	5.8	0.0	0.0	573.8	8.3	89.7	42.8	49.2	-1.8	
0.1	0.1	0.0	0.1	0.0	0.0	0.0	0.0	0.1	0.2	0.5	0.4	
5.56	96.43	-1.41	5.54	0	0	573.29	5.14	91.05	60.71	65.77	-0.86	
5.55	96.52	-1.41	5.52	0	0	573.28	5.12	91.28	60.97	66.09	-0.88	
5.3	96.56	-1.33	5.47	0	0	573.49	5.06	91.37	61.22	66.44	-0.97	
5.62	96.15	-1.33	5.5	0	0	573.5	5.12	91.37	61.41	66.53	-0.99	
5.6	96.18	-1.32	5.51	0	0	573.54	5.13	90.45	60.43	65.27	-1.01	
5.51	96.24	-1.38	5.49	0	0	573.37	5.11	90.59	60.6	65.49	-0.9	
5.5	96.3	-1.4	5.5	0.0	0.0	573.4	5.1	90.59	60.7	65.7	-0.9	
0.0	0.2	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.4	0.5	0.1	
4.84	96.57	-1.24	4.8	0	0	573.26	4.44	91.39	64.95	69.55	-0.73	
4.78	96.72	-1.17	4.75	0	0	573.43	4.4	91.78	65.51	70.18	-0.81	
4.79	96.74	-1.15	4.73	0	0	573.51	4.4	91.75	65.65	70.1	-0.84	
4.8	96.56	-1.22	4.76	0	0	573.29	4.4	91.37	65.14	69.7	-0.74	
4.82	96.65	-1.16	4.78	0	0	573.41	4.42	91.8	65.22	69.86	-0.8	
4.78	96.77	-1.19	4.71	0	0	573.39	4.36	91.68	65.79	70.45	-0.78	
4.8	96.7	-1.2	4.8	0.0	0.0	573.4	4.4	91.6	65.4	70.9	-0.8	
0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.2	0.3	0.4	
5.77	96.44	-1.4	5.79	0	0	573.46	5.34	91.08	59.64	64.8	-0.9	
5.75	96.55	-1.38	5.73	0	0	573.52	5.33	91.34	59.91	65.21	-1.03	
5.78	96.57	-1.41	5.76	0	0	573.46	5.35	91.39	59.79	65.13	-1	
5.64	96.53	-1.42	5.61	0	0	573.33	5.21	91.33	60.51	65.71	-0.9	
5.64	96.59	-1.38	5.61	0	0	573.44	5.21	91.43	60.83	65.87	-0.98	
5.63	96.57	-1.43	5.61	0	0	573.3	5.19	91.38	60.82	65.83	-0.98	
5.7	96.59	-1.4	5.7	0.0	0.0	573.4	5.3	91.3	60.2	65.4	-1.0	
0.1	0.1	0.0	0.1	0.0	0.0	0.0	0.1	0.1	0.5	0.6	0.1	
4.52	96.63	-1.18	4.47	0	0	573.3	4.13	91.54	66.81	71.16	-0.84	
4.49	96.78	-1.12	4.44	0	0	573.35	4.11	91.69	67.28	71.72	-0.72	
4.49	96.78	-1.1	4.44	0	0	573.45	4.11	91.89	67.28	71.71	-0.7	
4.52	96.77	-1.14	4.47	0	0	573.31	4.13	91.71	67	71.41	-0.7	
4.57	96.63	-1.17	4.52	0	0	573.26	4.18	91.55	66.54	70.98	-0.89	
4.59	96.59	-1.25	4.54	0	0	572.97	4.19	91.43	66.35	70.76	-0.95	
4.5	96.7	-1.2	4.5	0.0	0.0	573.3	4.1	91.77	66.8	71.3	-0.87	
0.0	0.1	0.1	0.0	0.0	0.0	0.0	0.2	0.0	0.2	0.4	0.1	
5.8	95.91	-0.84	5.8	0	0	575.06	5.52	89.78	58.37	63.21	-2.03	
5.78	95.99	-0.82	5.77	0	0	575.12	5.5	89.75	58.49	63.3	-2.06	
5.8	95.88	-0.87	5.8	0	0	574.99	5.52	89.72	58.32	63.14	-1.99	
5.8	95.98	-0.84	5.79	0	0	575.07	5.51	89.97	58.55	63.45	-2.03	
5.82	95.97	-0.86	5.81	0	0	575.02	5.53	89.93	58.41	63.31	-2.01	
5.85	95.95	-0.88	5.85	0	0	575.04	5.57	89.93	58.19	63.1	-2.04	
5.0	95.9	-0.8	5.0	0.0	0.0	575.1	5.5	89.8	58.4	63.3	-2.0	
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.1	

To Page No.

Witnessed & Understood by me,

Date \_\_\_\_\_

Invented by

*Alma*

Date \_\_\_\_\_

## EXHIBIT D

Project No. \_\_\_\_\_  
Book No. 14660TITLE Exp# 145 Brightness Results

From Page No. \_\_\_\_\_

Exp#	Sample#	side	position	Operator	TEST DATE	BRIGHTNESS	R(O)	R(O)	R(Z)	X	Y	Z	L	a
145	I	a	1	D	08/03/06	81.45	90.54	89.16	81.19	87.03	89.18	95	94.43	-0.8
145	I	a	2		08/03/06	81.47	90.63	89.16	81.21	87.03	89.18	96.01	94.42	-0.79
145	I	a	3		08/03/06	81.36	90.56	89.09	81.11	86.95	89.09	95.9	94.39	-0.81
145	I	b	1		08/03/06	81.46	90.77	89.27	81.18	87.13	89.27	95.96	94.45	-0.81
145	I	b	2		08/03/06	81.38	90.71	89.21	81.07	87.08	89.21	95.85	94.45	-0.83
145	I	b	3		08/03/06	81.36	90.73	89.21	81.08	87.08	89.21	95.86	94.45	-0.79
					Average	81.4	90.7	89.2	81.1	87.0	89.2	95.9	94.4	-0.8
					StdDev	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.0	0.0
145	J	a	1	D	08/03/06	80.46	90.06	88.51	80.19	86.39	88.51	94.81	94.08	-0.78
145	J	a	2		08/03/06	80.47	90.06	88.47	80.2	86.37	88.47	94.82	94.06	-0.77
145	J	a	3		08/03/06	80.33	89.95	88.38	80.07	86.27	88.38	94.67	94.01	-0.78
145	J	b	1		08/03/06	80.72	90.36	88.78	80.45	86.66	88.78	95.12	94.22	-0.78
145	J	b	2		08/03/06	80.59	90.27	88.68	80.3	86.57	88.58	94.84	94.17	-0.77
145	J	b	3		08/03/06	80.48	90.19	88.0	80.2	86.48	88.0	94.82	94.13	-0.79
					Average	80.5	90.2	88.6	80.2	86.5	88.6	94.9	94.1	-0.8
					StdDev	0.1	0.2	0.1	0.1	0.1	0.1	0.2	0.1	0.0
145	K	a	1	D	08/03/06	80.24	88.94	87.58	80	85.46	87.58	94.59	93.58	-0.8
145	K	a	2		08/03/06	80.3	88.99	87.59	80.06	85.51	87.59	94.65	93.59	-0.75
145	K	a	3		08/03/06	80.29	88.97	87.57	80.06	85.5	87.57	94.64	93.58	-0.74
145	K	b	1		08/03/06	79.49	87.99	86.63	79.25	84.57	86.63	93.7	93.07	-0.78
145	K	b	2		08/03/06	79.35	87.87	86.5	79.1	84.45	86.5	93.62	93	-0.75
145	K	b	3		08/03/06	79.33	87.86	86.5	79.08	84.43	86.5	93.5	93.01	-0.78
					Average	79.8	88.4	87.1	79.6	85.0	87.1	94.1	93.3	-0.8
					StdDev	0.5	0.6	0.5	0.5	0.6	0.6	0.6	0.3	0.0

To Page No. \_\_\_\_\_

Witnessed &amp; Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by Robert Riden

Recorded by \_\_\_\_\_

Date \_\_\_\_\_

## EXHIBIT D

TITLE Engo H-145 Brightness ResultsBook No. 14660

91

From Page No. \_\_\_\_\_

HUNTER W3TMACIE W CIE TINT													
b	L*	a*	b*										
5.91	95.65	-0.77	5.91	0	0	575.32	5.08	89.16	57.29	62	-2.28		
5.89	95.65	-0.76	5.9	0	0	575.34	5.05	89.16	57.36	62.05	-2.26		
5.92	95.62	-0.78	5.93	0	0	575.27	5.08	89.09	57.18	61.85	-2.22		
6	95.89	-0.78	6.01	0	0	575.3	5.75	89.27	56.88	61.89	-2.27		
6.04	95.67	-0.8	6.05	0	0	575.27	5.79	89.21	56.83	61.44	-2.27		
6.02	95.67	-0.78	6.03	0	0	575.38	5.78	89.21	56.7	61.5	-2.33		
6.0	95.7	-0.8	6.0	0.0	0.0	575.3	5.7	89.2	57.0	61.8	-2.3		
0.1	0.0	0.0	0.1	0.0	0.0	0.0	0.1	0.1	0.3	0.3	0.0		
6.19	95.37	-0.78	6.21	0	0	575.44	5.97	88.51	55.25	59.9	-2.45		
6.18	95.36	-0.74	6.18	0	0	575.48	5.95	88.47	55.38	60	-2.47		
6.19	95.32	-0.75	6.21	0	0	575.46	5.98	88.38	55.14	59.74	-2.47		
6.19	95.49	-0.75	6.21	0	0	575.48	5.97	88.78	55.46	60.21	-2.46		
6.23	95.45	-0.75	6.25	0	0	575.48	6.01	88.88	55.16	59.9	-2.49		
6.25	95.41	-0.76	6.27	0	0	575.44	6.03	88.6	55	59.72	-2.48		
6.2	95.4	-0.8	6.2	0.0	0.0	575.5	6.0	88.6	55.2	59.9	-2.5		
0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.2	0.0		
5.66	94.98	-0.77	5.67	0	0	575.22	5.48	87.56	57.32	61.34	-2.1		
5.63	94.99	-0.73	5.65	0	0	575.34	5.44	87.59	57.46	61.48	-2.17		
5.63	94.98	-0.71	5.65	0	0	575.37	5.44	87.57	57.47	61.49	-2.19		
5.54	94.58	-0.73	5.57	0	0	575.29	5.38	86.63	57.14	60.8	-2.12		
5.67	94.53	-0.72	5.6	0	0	573.33	5.41	86.5	56.9	60.53	-2.15		
5.59	94.53	-0.78	5.61	0	0	575.23	5.43	86.5	56.81	60.46	-2.1		
5.6	94.8	-0.7	5.6	0.0	0.0	578.3	5.4	87.1	57.2	61.8	-2.1		
0.0	0.2	0.0	0.0	0.0	0.0	0.1	0.0	0.6	0.3	0.5	0.0		

To Page No. \_\_\_\_\_

Witnessed &amp; Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Recorded by \_\_\_\_\_

Date \_\_\_\_\_

## EXHIBIT E

Project No.

Book No. 14500

TITLE

Expt 145

FAO

Results

From Page No.

Test	Field #	Sample	Temp	Flow	Half	Manufac	Manufac	Operator	Lab	Run	Dry Bulk	Dry Bulk	Wick	Wick	Wick Bulk	Wick Bulk	Capacity
				Type	Cond	Date	Time			Number	ccg	2.00Pa	Time	Rate	2.00Pa	0.04Pa	Capacity
											ccg	sec	mm/s	ccg	ccg	ccg	g/s
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	40.84	24.38	2.8	8.53	9.85	11.74	12.92
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	36.97	33.17	2.4	10.65	9.63	11.49	11.59
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	40.82	24.32	2.5	10.62	9.75	11.56	12.06
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	AV	40.11	23.89	2.57	10.77	9.76	11.59	11.9
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	40.84	24.32	2.1	12.93	10.52	12.26	12.62
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	40.84	24.32	2.8	10.4	10.46	12.37	12.55
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	40.82	24.38	2.37	11.53	10.49	12.28	12.54
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	41.03	26.38	3	10.96	10.73	18.74	16.55
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	42.03	26.61	3.2	10.23	10.59	18.56	18.82
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	42.03	26.61	3.03	10.62	10.53	18.48	18.59
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.7	26.18	3.1	10.48	15.53	18.48	18.58
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.87	26.76	3.3	10	10.59	18.48	18.53
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	41.78	29.29	2.8	10.79	14.68	17.3	18.08
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	43.38	26.06	3.1	10.42	15.24	18.39	18.53
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	41.85	25.22	2.4	12.87	13.8	16.75	16.8
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	42.38	25.81	2.8	11.13	14.38	17.28	17.61
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.37	25.33	2.6	10.64	14.35	17.18	17.37
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	42.27	25.39	2.7	11.48	14.14	17.03	17.25
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	42.1	25.24	2.9	10.8	15.22	18.03	18.19
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.84	25.8	2.9	10.88	15.28	18.1	18.42
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.08	25.87	3	10.73	15.23	18.18	18.38
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	41.77	24.84	2.6	11.5	13.73	18.49	18.88
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	44.16	25.87	2.8	11.95	14.31	17.27	17.17
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.16	25.87	2.7	11.43	14.12	16.94	16.95
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.48	2.3	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.87	25.28	2.33	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.72	25.04	2.3	11.65	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.87	2.3	11.41	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	41.77	24.84	2.6	11.5	13.73	18.49	18.88
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.16	25.87	2.8	11.95	14.31	17.27	17.17
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.48	2.3	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.87	25.28	2.33	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.72	25.04	2.3	11.65	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.87	2.3	11.41	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	41.77	24.84	2.6	11.5	13.73	18.49	18.88
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.16	25.87	2.8	11.95	14.31	17.27	17.17
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.48	2.3	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.87	25.28	2.33	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.72	25.04	2.3	11.65	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.87	2.3	11.41	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	41.77	24.84	2.6	11.5	13.73	18.49	18.88
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.16	25.87	2.8	11.95	14.31	17.27	17.17
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.48	2.3	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.87	25.28	2.33	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.72	25.04	2.3	11.65	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.87	2.3	11.41	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	41.77	24.84	2.6	11.5	13.73	18.49	18.88
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.16	25.87	2.8	11.95	14.31	17.27	17.17
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.48	2.3	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.87	25.28	2.33	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.72	25.04	2.3	11.65	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.87	2.3	11.41	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	41.77	24.84	2.6	11.5	13.73	18.49	18.88
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.16	25.87	2.8	11.95	14.31	17.27	17.17
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.48	2.3	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.87	25.28	2.33	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.72	25.04	2.3	11.65	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.87	2.3	11.41	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	41.77	24.84	2.6	11.5	13.73	18.49	18.88
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.16	25.87	2.8	11.95	14.31	17.27	17.17
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.48	2.3	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.87	25.28	2.33	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.72	25.04	2.3	11.65	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.87	2.3	11.41	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	41.77	24.84	2.6	11.5	13.73	18.49	18.88
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.16	25.87	2.8	11.95	14.31	17.27	17.17
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.48	2.3	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.87	25.28	2.33	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.72	25.04	2.3	11.65	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.87	2.3	11.41	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	41.77	24.84	2.6	11.5	13.73	18.49	18.88
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	44.16	25.87	2.8	11.95	14.31	17.27	17.17
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	3	43.08	25.48	2.3	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	1	42.87	25.28	2.33	11.84	14.35	16.93	16.93
FA	14500	exp145	Adm145	O	A	8/12/06		dr	Lab 118	2	43.72	25.04	2.3	11.65	14.35	16.93	16.93